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IS 12308-4 (1988): Methods for Chemical Analysis of Cast Iron and Pig Iron, Part 4: Determination of Total Carbon, Graphitic Carbon and Combined Carbon by Gravimetric Method [MTD 6: Pig iron and Cast Iron]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHODS FOR
CHEMICAL ANALYSIS OF CAST IRON
AND PIG IRON**

**PART 4 DETERMINATION OF TOTAL CARBON, GRAPHITIC CARBON
AND COMBINED CARBON BY GRAVIMETRIC METHOD**

भारतीय मानक

ढलवां लोहे और कच्चे लोहे के रासायनिक विश्लेषण की पद्धतियां

भाग 4 भारात्मक पद्धति द्वारा कुल कार्बन, ग्रेफाइटिक कार्बन और मिश्रित कार्बन ज्ञात करना

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FOREWORD

This standard (Part 4) was adopted by the Bureau of Indian Standards on 17 October 1988, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

Chemical analysis of cast iron and pig iron was earlier covered in IS 228 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (*revised*)'. During its second revision, it was decided that separate comprehensive series should be prepared, one for chemical analysis of all types of steels and the other covering chemical analysis of pig iron and cast iron. Accordingly, IS 228 on revision was published in several parts covering chemical analysis of various steels only, and a separate series of standard under IS : 12308 is being published to cover chemical analysis of cast iron and pig iron. This standard (Part 4) is one in the latter series. The other parts in the series are:

IS : 12308 Methods for chemical analysis of cast iron and pig iron

Part 1 Determination of total carbon by thermal conductivity method

Part 2 Determination of sulphur by iodimetric titration after combustion

Part 3 Determination of manganese by periodate spectrophotometric method

The method given in Part 4 has been prescribed on the basis of inter-laboratory tests carried by the various laboratories on standard samples of the respective materials.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 4 DETERMINATION OF TOTAL CARBON, GRAPHITIC CARBON AND COMBINED CARBON BY GRAVIMETRIC METHOD

1 SCOPE

1.1 This standard (Part 4) covers the gravimetric method for determination of total carbon, graphitic carbon and combined carbon in cast iron, alloyed cast iron and pig iron.

2 REFERENCE

IS 6626 (Part 1) : 1971 Recommendations for apparatus for chemical analysis of metals; Part 1. Determination of carbon by direct combustion, is a necessary adjunct to this standard.

3 SAMPLING

3.1 The sample shall be drawn and prepared as prescribed in the relevant Indian Standard.

3.2 The sample is to be cleaned with analar grade organic solvent (like acetone, benzene or ether) by washing it thrice and dried in an air oven at $100 \pm 5^\circ\text{C}$.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5 DETERMINATION OF TOTAL CARBON, GRAPHITIC CARBON AND COMBINED CARBON BY GRAVIMETRIC METHOD

5.1 Outline of the Methods

5.1.1 Total Carbon

The sample is burnt in the presence of oxygen. The carbon dioxide formed is absorbed in a tube containing ascarite or soda asbestos and weighed.

5.1.2 Graphitic Carbon

The sample is dissolved in nitric acid and hydrofluoric acid. The graphitic carbon is filtered off in preignited asbestos in a fused silica gooch crucible and dried and ignited in a combustion furnace. Carbon dioxide so formed is absorbed in a tower of soda aspirate and weighed.

5.1.3 Combined Carbon

The difference between the percentage of total carbon and graphite carbon represents the combined carbon.

5.2 Reagents

5.2.1 Tower Packed with Granulated Manganese Dioxide

5.2.2 Tower Packed with Magnesium Perchlorate

5.2.3 Absorption Tower Packed with Soda Aspirate and Magnesium Perchlorate

5.2.4 Dilute Nitric Acid, 1 : 2 (v/v)

5.2.5 Hydrofluoric acid, 40 percent

5.2.6 Preignited Asbestos for Gooch Crucible, (preserved in a desiccator)

5.3 Apparatus

The apparatus recommended in IS 6626 (Part 1) : 1971 Recommendations for apparatus for chemical analysis of metals; Part 1 Determination of carbon by direct combustion may be used.

5.4 Procedure

5.4.1 For Total Carbon

Weigh 1.00 g of sample on a preignited combustion boat and introduce the same into the combustion tube maintained at a temperature of 1100°C or above.

Stopper the combustion tube inside the furnace and allow the sample to heat for 1 to 2 minutes. Then pass oxygen at the rate of 800-1000 ml per minute while combustion is going on and allow the evolved gases to pass through the tower of granular manganese dioxide and magnesium perchlorate and finally to the absorption tower (5.2.3) (tube A) kept in series. When the combustion is complete (about 2 minutes), reduce the rate of flow of oxygen to 400-500 ml/minute and continue for another 7-8 minutes in order to sweep off the carbon dioxide. Withdraw the absorption tower (tube A) filled with oxygen and place it in the balance case for 10 minutes,

open momentarily and weigh again and note the increase in mass of tube *A*.

Make a blank determination, following the same procedure and using the same amounts of all reagents except the sample.

5.4.1.1 Calculation

Total carbon

$$(\text{percent}) = \frac{(A - B) \times 0.2729 \times 100}{C}$$

where

A = increase in mass (g) with the sample,

B = increase in mass (g) without the sample, and

C = mass in g of sample taken.

5.4.2 For Graphitic Carbon

Dissolve 1.00 g of sample in 50 ml of dilute nitric acid and heat on a water bath for 30 minutes. Add 1 to 2 drops of hydrofluoric acid and boil for 4 to 5 minutes. Remove from the

water bath and collect the residue by suction on ignited asbestos contained in fused silica Gooch crucible. Wash thoroughly with hot water, dry the residue at 150°C and determine the graphitic carbon by direct combustion at 1 000°C.

5.4.3 For Combined Carbon

The difference between total carbon and the graphitic carbon content represents the percentage of combined carbon. In case of alloyed cast iron, it is convenient to use a flux, such as red lead, or tin or vanadium pentoxide (free from carbon) to enhance the combustion.

5.5 Reproducibility

- a) For total carbon
 - ± 0.07 at 2 percent carbon, and
 - ± 0.1 at 4 percent carbon.
- b) For graphitic carbon
 - ± 0.20 at 1 percent carbon, and
 - ± 0.25 at 2 percent carbon.

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